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Cadmium Oxide Nanoparticles as A Novel Photo-Catalyst for Degradation of Ciprofloxacin Antibiotic in Aqueous Media

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Abstract

In the present work, the cadmium oxide (CdO) nanoparticles (NPs) are synthesized using the Ferula extract. Ferula acts as a naturally-sourced reducing agent and stabilizer for the construction of the CdO NPs. The biosynthesized CdO NPs are characterized by different techniques such as X-ray powder diffraction (XRD), Fourier transform-infrared (FT-IR), spectroscopy and field emission-scanning electron microscopy (FE-SEM). After ensuring a successful synthesis of the CdO NPs, their photocatalytic activity is studied for the degradation of ciprofloxacin antibiotic in aqueous media under the sunlight. Approximately 95% degradation of ciprofloxacin using the CdO NPs is achieved after 60 minutes. The recycling experiments confirm the high stability and durability of the CdO NPs. Therefore, this work illustrates an efficient strategy for the photo-degradation of ciprofloxacin, and provides a new insight into the removal of pharmaceutical contaminants in aquatic environments.

1. Introduction

The recent public attention to the presence of organic contaminants in wastewater has raised important concerns [1]. The organic contaminants, especially antibiotics, can disrupt the natural life of plants and animals, and even the humans [2]. One of the most devoted antibiotics is the fluoroquinolones (FQs), which are applied for the treatment of bacterial infections [3]. Ciprofloxacin (CIP) is a second-generation fluoroquinolone [4, 5]. It is utilized medically in order to treat the infections of urinary tract, skin, and genitals [6]. CIP enters the aquatic environments through the excretion of humans and animals [7]. Unfortunately, the widespread presence of CIP in water may lead to the development and spread of the antibiotic-resistant bacteria [8], which according to the World Health Organization (WHO), is one of the three major threats to the human health. Due to the importance of this issue, various techniques such as adsorption, sedimentation, electrochemical reduction, coagulation, membrane filtration, and chemical oxidation have been introduced in order to remove drug contaminants [9, 10]. Despite the significant advantages each one may have, these techniques have drawbacks in the practical environmental remediation including the transfer of contaminants from the liquid to the solid phase, the need for hazardous chemicals, generation of sludge, and requirement for further treatment processes and complex equipment. Also the conventional treatment methods are inefficient in removing the contaminants in trace levels. Therefore, it is indispensable to use an effective technique to remove CIP in aquatic media.

Recently, the photo-catalytic processes using metal oxide semi-conductors have been known as a safe alternative to the traditional approaches of

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water treatment [11-13]. In the presence of a semi-conductor and the light source, electrons and holes are formed that can activate H_2O and O_2 in order to produce reactive oxygen species [14]. Ultimately, these active species lead to the elimination of pollutants without the need for complex equipment.

The cadmium oxide (CdO) nanoparticles (NPs) are an n-type semi-conductor that are a suitable candidate for various technological applications in a variety of fields [15-19]. Due to their unique properties, several synthetic processes such as hydrothermal, mechanochemical milling, microwave-assisted, sol-gel, and solvothermal and sonochemical methodologies have been reported for the fabrication of the CdO NPs [20-24]. Most of these methods are complex and time-consuming, and generate harmful waste. Relying on the environmental protection, the green synthesis of the CdO NPs using plant extracts is favored [25, 26]. This approach has the advantages of producing minimal waste, and no usage of harmful reducing agents, toxic solvents, and expensive stabilizers.

Ferula persica, the well-known *Ferula*, belongs to the Apiaceae family [27]. *Ferula* is endemic to the Iran, Turkey, and Afghanistan countries. *Ferula* persica is commonly called Koma in Iran. *Ferula* is an herbaceous, hairy, perennial plant with fleshy and slightly thick roots and strong and rough stems. It has been used in traditional medicine for the treatment of diseases such as diabetes, backache, rheumatism, gout, and sinusitis due to their antihypertensive, anti-inflammatory, anti-tumour and anti-angiogenic properties [28-30]. *Ferula* is a rich source of the phenolic compounds [31, 32]. Since the phenolic compounds are reducing agents of metal ions, therefore, *Ferula* is a suitable candida for the green synthesis of nanoparticles [28, 33, 34].

Inspired from this green approach, in the present work, the CdO NPs were synthesized using the *Ferula* plant and characterized. To the best of the authors' knowledge, this is the first report for the green synthesis of the CdO NPs using *Ferula*. In the following, due to the successful results of the CdO NPs in the removal of organic contaminants [35-38], inhere, the catalytic performance of the CdO NPs was investigated for the photo-degradation of CIP in an aqueous solution under the sunlight. The main objectives of this work were: (1) fabrication of the CdO NPs *via* a green route and without the use of toxic solvents and expensive reducing agents; (2) evaluation of the catalytic performance of the biosynthesized catalyst for degradation of an antibiotic; and (3) checking the reusability and stability of the CdO NPs. Hence, it helps to provide a practical reference in the development of a nanocatalyst for degradation of the other antibiotics in wastewater treatment.

2. Experimental Section

2.1. Materials

Cadmium nitrate tetrahydrate (99+%) was purchased from the ACROS company. The antibiotic was supplied by the Farabi pharmaceutical company (Iran).

2.2. Characterization

XRD measurement was done using a Philips X'pert diffractometer type PW 1800 goniometer (Cu Kα = 1.5406 A°). Fourier transform-infrared (FT-IR) spectrum of the CdO NPs was recorded using the FT-NIR spectroscope (RAYLEIGH, WQF-510). The morphology of the biosythesized CdO NPs was studied by FE-SEM (Mira 3-XMU). The concentration of the antibiotic was measured using a UV–visible spectrophotometer (GENWAY).

2.3. Preparation of *Ferula* extract

The *Ferula* plant was collected from the plains of the South Khorasan province in Iran. The *Ferula* plant was rinsed with water for several times for the removal of dust particles, shadow-dried for two weeks, and finally, powdered using a mixer grinder. Then 10 g of the *Ferula* powders in 100 mL of deionized water was refluxed at 80 °C for 20 min. The extract of *Ferula* was filtered using the Whatman No. 1 filter paper, and utilized for the biosynthesis of the CdO NPs.

2.4. Biosynthesis of CdO NPs using *Ferula* extract

50 mL of the *Ferula* plant extract was drop-wisely added to 50 mL of a well-mixed 0.01 M aqueous solution of $Cd(NO_3)_2.4H_2O$ at the laboratory temperate. Stirring of the solution was continued for another 2 hours at 80 °C. The color of the solution was changed from colorless to brown due to excitation of the surface plasmon resonance. This phenomenon indicated the formation of the CdO NPs and the hydrogen donation activity of the phenolic compounds inside the plant. The sediment formed was rinsed for five times with water, and then dried at the laboratory temperature. Finally, the obtained powder was

collected in a ceramic crucible, and heated in an electric furnace at 500 °C for 2 hours.

2.5. Photo-degradation of ciprofloxacin using CdO NPs under sunlight

For studying the photocatalytic activity of the CdO NPs, the tests were accomplished under the sunlight on September 2021 (latitude 32.8621 and longitude 59.1939). Briefly, 50 mg of the CdO NPs was added to 50 mL of the CIP solutions (10 ppm), and then stirred in the darkness for 30 min to reach the adsorption–desorption equilibrium. During the experiments, 2.0 mL of the suspension was taken out at given time intervals and separated using filter papers to remove the CdO NPs. The degradation efficiency of ciprofloxacin was calculated as below:

$$DE(\%) = \frac{(C_0 - C)}{C_0} \times 100$$

where DE was defined as the degradation efficiency of ciprofloxacin, and C_0 and C denote the initial and final concentrations of ciprofloxacin.

3. Results and Discussion

3.1. Fabrication and characterization of CdO NPs

To the best of our knowledge, the biosynthesis of the CdO NPs was done using the *Ferula* plant for the first time in this work. The image of the *Ferula* plant is exhibited in Figure 1.

The phenolic compounds present in the *Ferula* plant are responsible for the bioreduction of Cd^{2+} ions to the Cd NPs (Figure 2). *Ferula* in addition to the role of reducing agent, effectively prevents the agglomeration of the Cd NPs. The Cd NPs convert to the CdO NPs by calcination in furnace at 500 °C.

Figure 1. *Ferula* plant.

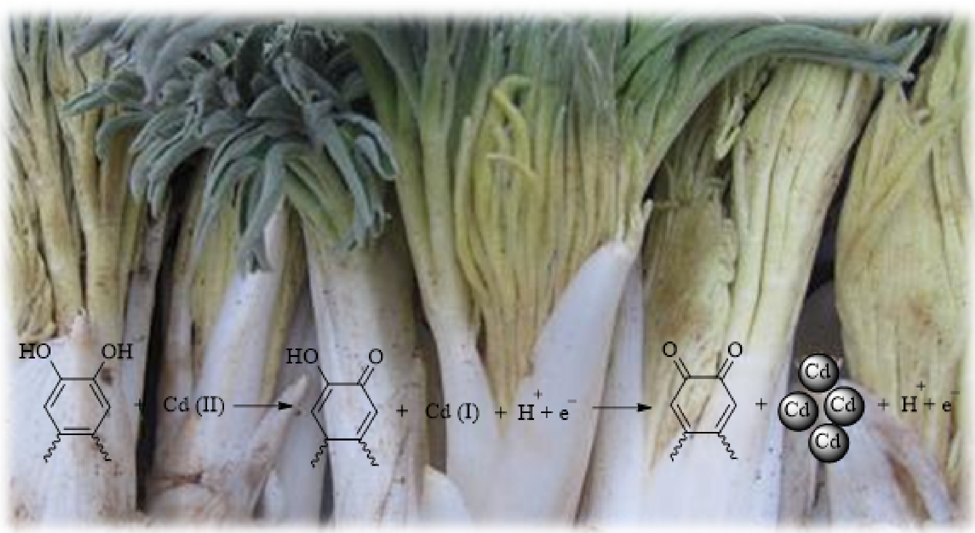


Figure 2. Proposed mechanism for green synthesis of Cd NPs.

The XRD pattern of the CdO NPs is displayed in Figure 3. The positions of the diffraction peaks are seen at 2θ = 33.01, 38.30, 55.29, 65.90, and 69.27, corresponding to the (111), (200), (220), (311), and (222) planes of the CdO NPs, respectively. The XRD pattern of the biosynthesized NPs matches with the cubic Monteponite CdO structure (JCPDS 05-0640) [16]. The crystallite size of the biosynthesized CdO NPs was calculated using the Debye-Scherrer equation [39], and found to be 41 nm.

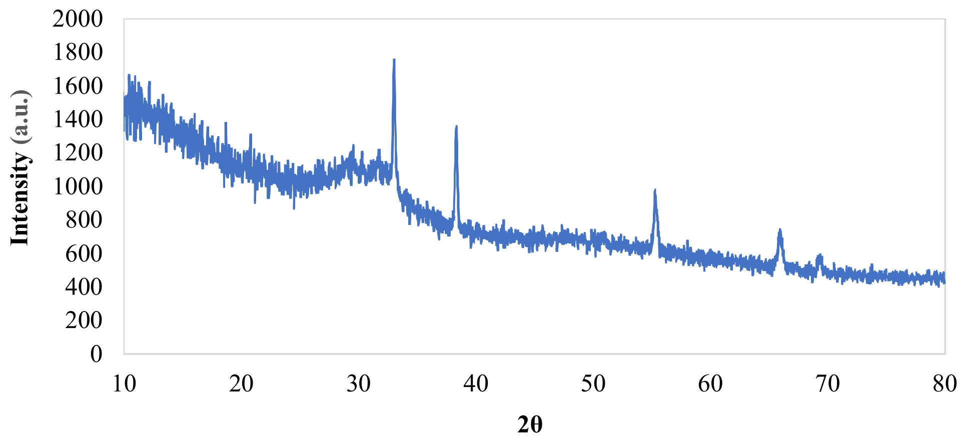


Figure 3. XRD pattern of synthesized CdO NPs using *Ferula*.

The FT-IR analysis was utilized to investigate the functional groups and purity of the biosynthesized CdO NPs (Figure 4). The FT-IR spectrum displays a broad absorption band at 3433 cm^{-1} due to the stretching vibrations of the O-H group [40]. The absorption bands at 2912 cm^{-1} and 1022 cm^{-1} is attributed to the stretching vibrations of C–H and C-O functional group in extract, respectively [41]. The absorption peak at 547 cm^{-1} is related to the stretching vibrations of Cd–O [42]. The presence of this band indicates the successful fabrication of the CdO NPs.

The shape and morphology of the CdO NPs were studied using the FE-SEM technique. As it can be seen in Figure 5, the CdO NPs has a polygonal structure with an almost uniform size distribution.

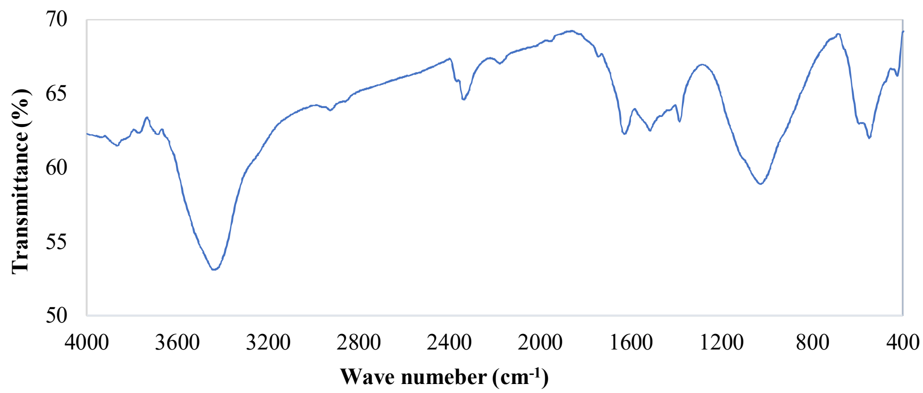


Figure 4. FT-IR spectrum of biosynthesized CdO NPs using *Ferula*.

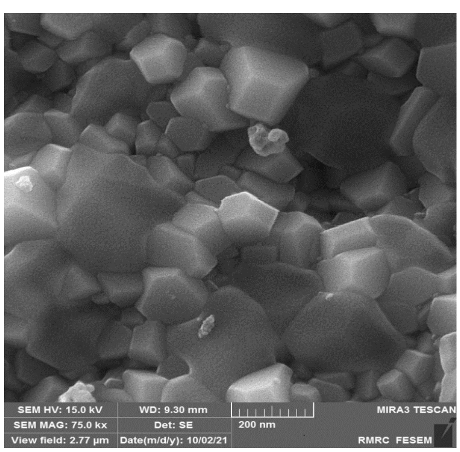


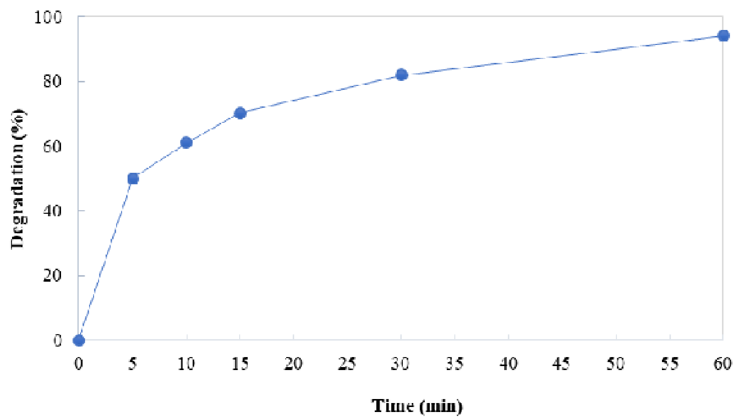
Figure 5. FE-SEM micrograph of synthesized CdO NPs using *Ferula*.

3.2. Evaluation of photocatalytic performance of CdO NPs for degradation of ciprofloxacin under sunlight

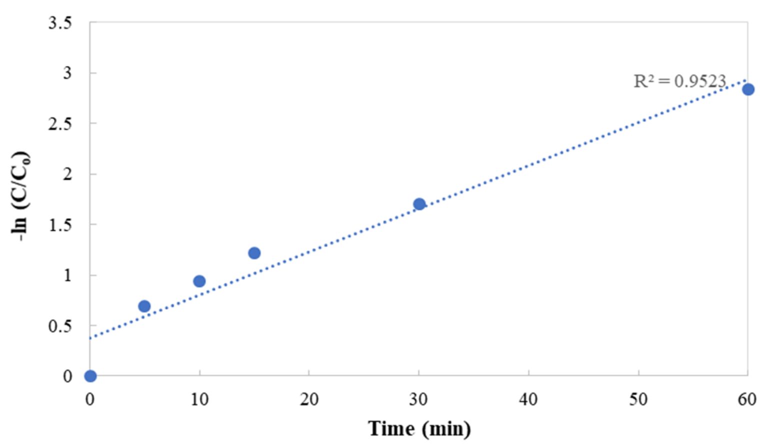
The photocatalytic performance of the CdO NPs was examined by the degradation of ciprofloxacin in aqueous solution under the sunlight. The change in concentration of the ciprofloxacin solution during the photocatalytic process was monitored using UV–visible spectroscopy at the wave length CA. 275 nm [43]. As shown in Figure 6a, 94.12% of ciprofloxacin was effectively degraded after 60 min. No other characteristic peak was observed during the degradation process. This result obtained indicate that the ciprofloxacin antibiotic in the presence of the CdO NPs is directly mineralized into carbon dioxide and water or effectively degrades into other smaller products. In order to better demonstrate the photocatalytic performance of the CdO NPs, the degradation of ciprofloxacin was investigated in the absence of the CdO NPs under the sunlight. Any degradation of ciprofloxacin was not shown in the absence of photo-catalyst after 60 min. The rate constant of the degradation of ciprofloxacin using the CdO NPs was calculated through the following equation:

$$-\ln\left(\frac{C}{C_0}\right) = k_{obs}t$$

where C is the concentration of ciprofloxacin (ppm) at time t (min), C_o is the initial concentration of ciprofloxacin, and k_{obs} is the pseudo-first-order rate constant (min^{-1}). Figure 6b displays the curve of $-\ln\left(\frac{C}{C_0}\right)$ *vs.* t, exhibiting a linear trend and indicating that the ciprofloxacin degradation is fitted to the pseudo-first-order kinetic model. This result is consistent with the reports in the literature [44]. Also k_{obs} was calculated as 0.04722 min^{-1}.



(a)



(b)

Figure 6. (a) Degradation efficiency and (b) pseudo-first-order kinetic fitting of ciprofloxacin using CdO NPs under sunlight.

In practice, the recyclability and stability of catalysts can greatly reduce the cost of water treatment and prevent the secondary contamination. In order to evaluate the stability of the biosyntheized photo-catalyst, after each cycle of reaction, the CdO NPs were separated from the reaction mixture by filter paper, and then rinsed with water for five times and then dried. In the next cycle, the CdO NPs were reused for photo-degradation of the fresh antibiotic solution. The result obtained showed that after three cycles, the photo-catalytic activity of the CdO NPs did not show a significant decline.

In order to demonstrate the superiority of the CdO NPs photo-catalytic performance compared to the other photo-catalysts for ciprofloxacin degradation, some previous reports are compared in Table 1. As revealed in this table, the CdO photo-catalyst was synthesized *via* a green route without the need for harmful chemical reagents and hazardous organic solvents in this work. Also the CdO NPs show a more efficient performance than the other catalysts for photo-degradation of ciprofloxacin. Most importantly, the presence of sunlight as a renewable and inexpensive radiation source is one of the main advantages of this method.

Table 1. Comparison of removal efficiency of ciprofloxacin.

Catalyst	Catalyst synthesis method	Source of stimulation	Degradation time (min)	Efficiency (%) [Ref]
N,S Co-doped TiO_2	Sol–gel	UV–visible light	150	78.7 [45]
TiO_2/MMT	Hydrothermal	Ultrasonic	120	65 [46]
$Bi_7(PO_4)O_9$	Sonochemical	Simulated solar light	120	91 [47]
Ag_3PO_4	Precipitation deposition	Xenon lamp	120	87 [48]
CeO_2–Ag/AgBr	Co-precipitation	Xenon lamp	120	93.05 [49]
TiO_2	-*	UV lamp	120	91 [50]
$ZnWO_4$-CdS	Hydrothermal	Xenon lamp	60	83 [51]
CdO NPs	Green	Natural sunlight	60	94.12 [This work]

* TiO_2 was purchased.

4. Conclusions

The present work expressed a simple method for the biosynthesis of the CdO NPs using the *Ferula* plant, acting as an efficient reducing agent and stabilizer. In this synthetic process, the natural, eco-friendly, and renewable materials were used without the need for expensive or harmful chemical reagents and hazardous organic solvents. The phase identification of the CdO NPs was investigated using XRD, and the crystallite size of the biosynthesized NPs was calculated using the Debye-Scherrer equation, and found to be 41 nm. The presented functional groups in the biosynthesized NPs were identified by FT-IR spectral analysis, and the absorption peaks indicated the successful synthesis of the CdO NPs. The shape and morphology of the CdO NPs were studied using the FE-SEM technique, and specified that the biosynthesized NPs had a polygonal structure without no noticeable aggregation. After ensuring the successful synthesis of the biosynthesized NPs, the CdO NPs were employed as an efficient photo-catalyst for the degradation of ciprofloxacin under the sunlight. The results obtained showed that the photo-catalytic degradation of ciprofloxacin using the CdO NPs followed the pseudo-first-order kinetic. Moreover, the CdO NPs could be readily reused for at least three times without any significant loss in their catalytic performance. The remarkable advantages of this protocol include a simple and safe method for the synthesis of the CdO NPs, high yield of degradation, and recyclability of the catalyst. Most importantly, using the sunlight as an inexpensive and renewable energy source is one of the main advantages of this method.

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نانوذرات اکسید کادمیوم به عنوان یک فوتوکاتالیست جدید برای تخریب آنتی بیوتیک سیپروفلوکساسین در محیط‌های آبی

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چکیده:

در تحقیق حاضر، نانوذرات (NPs) اکسید کادمیوم (CdO) با استفاده از عصاره فرولا سنتز شدند. فرولا به عنوان یک عامل کاهنده و پایدارکننده طبیعی برای ساخت نانوذرات CdO عمل می‌کند. نانو ذرات CdO بیوسنتز شده با تکنیک‌های مختلفی مانند پراش پودر پرتو ایکس(XRD)، طیف‌سنجی مادون قرمز تبدیل فوریه(FT-IR) و میکروسکوپ الکترونی روبشی نشر میدانی (FE-SEM) شناسایی شد. پس از اطمینان از سنتز موفقیت آمیز نانوذرات CdO، فعالیت فوتوکاتالیستی آن‌ها برای تخریب آنتی‌بیوتیک سیپروفلوکساسین در محیط‌های آبی زیر نور خورشید مورد مطالعه قرار گرفت. پس از ۶۰ دقیقه حدود ۹۵٪ تخریب سیپروفلوکساسین با استفاده از نانوذراتCdO حاصل شد. آزمایش‌های بازیافت، پایداری و دوام بالای نانوذرات CdOرا تایید کردند. بنابراین، این کار یک استراتژی کارآمد برای تخریب نوری سیپروفلوکساسین را نشان داد، که بینش جدیدی را در مورد حذف آلاینده‌های دارویی در محیط‌های آبی ارائه نمود.

کلمات کلیدی: اکسید کادمیوم، نانوذرات، فرولا، فوتوکاتالیست، آنتی بیوتیک سیپروفلوکساسین.